1,147,210

PATENT SPECIFICATION 1,147,210

NO DRAWINGS.

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COMPLETE SPECIFICATION

Improvements in or relating to Vitamins.

We, EASTMAN KODAK COMPANY, a Company organized under the Laws of the State of New Jersey, United States of America, of 343, State Street, Rochester, New York 14650, United States of America (Assignee of LAWRENCE ANTHONY ANDERSON), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to vitamin products.

The early development of dry, finely divided, solid, fat-soluble vitamin-active products was based on a water gelling colloid such as gelatin as the vehicle or carrier for the fat-soluble vitamin-active oil. A process, referred to as a polyphase dispersion process, was developed for making such products. It is disclosed in such patents as the U.S. Patent, No. 2,183,053, to Taylor and the U.S. Patent, No. 2,183,084, to Reynolds. This process comprises the steps of preparing a warm emulsion of the fat-soluble vitamin-active oil in an aqueous, colloidal solution of the water gelling colloid, dispersing the emulsion into droplets in a water immiscible liquid such as mineral oil, and cooling the droplets to a temperature at which the colloid gels, whereby moist, solid particles or beadlets are formed. In one embodiment of the polyphase dispersion process the particles thus formed are separated by filtration and subsequent solvent washing from the water immiscible liquid, and then air dried as by fluid bed air drying. Because the gelled particles maintain their spherical shape after removal of the supporting, water immiscible liquid, they can be easily separated from the water immiscible liquid by filtration and they can be dried without continuous mechanical support for each gelled particle. In another embodiment of the process water is extracted from the solid particles by a water extraction solvent either after separation of the particles from the water immiscible liquid or while the particles are in the water immiscible liquid, and subsequent air drying can also be employed. In this embodiment gelation of the particles prior to dehydration minimizes in each particle outward diffusion of the fat-soluble vitamin-active oil and inward diffusion of the water extraction solvent.

In most instances, water gelling colloids and thus beadlet products made from them are not cold water dispersible or soluble. On the other hand, colloids which have the property of cold water dispersibility generally do not gel in water and do not possess the beadlet making advantages that water gelling colloids like gelatin offer.

Consequently, this has led to the development of processes, different from the polyphase dispersion process, for making dry, finely divided, solid, fat-soluble vitamin-active products wherein the carrier for the fat-soluble vitamin-active oil is a colloid. In these processes the transition from the liquid state to the solid state of the particles is accomplished in one of the following ways: (a) by very rapidly drying droplets of emulsion falling in a heated air stream, that is, spray drying, (b) by giving complete mechanical support to the emulsion during drying, for example, by drum drying, and (c) converting the emulsion to a self-supporting solid or semi-solid condition prior to or during its dehydration.

The spray drying process provides spherical beadlet type products, but at its present stage of development, these products apparently cannot be made from nongelling colloid emulsions with a sufficiently good yield in the −30+120 mesh, U.S. screen

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size, range or with a sufficiently high density (above 45 pounds per cubic foot).

A mechanical support process is disclosed in Patent No. 824,912. In this patent the process is directed to the preparation of a dry, finely divided, solid, vitamin E product wherein the carrier is acacia. The process comprises the steps of preparing an emulsion of an oily concentrate of Vitamin E active material in a colloidal solution of acacia and water, and dehydrating the emulsion with the aid of a complete mechanical support, that is, dehydrating the emulsion by spreading it out on a drying surface furnished by a heated drum dryer.

An example of a process wherein the emulsion is converted to a self-supporting solid or semi-solid condition prior to or during its dehydration, a thickening process, is disclosed in Patent No. 1,002,449. Here, the emulsion, based on acacia as the nongelling colloid, is converted to a rubbery dough by the addition of more acacia, the dough is formed into a thin shape, and the thin shaped dough is then dried.

Both the mechanical support process and the thickening process have certain limitations. For example, products with a high bulk density apparently cannot be made by the process of Patent No. 824,912 because the emulsion is dehydrated very rapidly and this gives rise to a porous structure in the product particles. The thickening process of Patent No. 1,002,449 provides a product with a high bulk density, but requires specialized, complex equipment. Both processes require comminution of the dehydrated product, which results in fragmented particles, substantial percentages of fines and yields of product in various particle size ranges less than desired.

There is a need, therefore, for a practical process for making a dry, solid, fat-soluble vitamin-active product based on nongelling colloid material, which avoids the disadvantages of the spray drying process, the mechanical support process and the thickening process.

This invention is based upon the discovery that under certain critical conditions, all spherical beadlets of a nongelling colloid containing dispersed therein a fat-soluble vitamin-active material can be prepared by a process comprising a polyphase dispersion procedure.

In summary, this invention comprises a process for making a dry free-flowing, finely divided, fat-soluble vitamin-active product wherein cold water dispersible, non-gelling colloid material is employed as the carrier for a water insoluble, fat-soluble vitamin-active composition. This process comprises: (1) preparing a colloidal solution of said colloid material and water; (2) dispersing in said colloidal solution said water insoluble, fat-soluble vitamin-active composition, whereby a first dispersion is formed; (3) dispersing said first dispersion in a water immiscible dispersing medium, whereby a second dispersion is formed; (4) extracting at a temperature in a range from −10 to 0°C. with a water extracting agent water from said second dispersion until said colloidal material solidifies, whereby finely divided, solid particles containing said water insoluble, fat-soluble vitamin-active composition dispersed therein are formed; (5) separating at a temperature in a range from −10 to 0°C. solid particles from said dispersing medium and (6) removing substantially all residual moisture from said solid particles.

In preferred embodiments of this invention lecithin is incorporated into said second dispersion for the purpose of stabilizing said second dispersion during the dehydration step and to maintain the sphericity of the dispersed droplets of said first dispersion during the dehydration step. Although the lecithin can be incorporated into the second dispersion by adding it to the first dispersion and by adding it to the water immiscible dispersing medium prior to adding the first dispersion thereto, it is preferred for best results that the lecithin be incorporated into the second dispersion by adding it at the same time as the water extracting agent is added.

The cold water dispersible, nongelling colloid material is cold water dispersible material which does not gel in water under normal conditions. It includes nongelling, cold water dispersible, naturally occurring gums such as acacia (sometimes referred to as gum arabic) and gum ghatti, purified amylopectin, pregelatinized and thin boiling starches, substituted starches such as starch esters, for example, corn starch which has been partially reacted with octenyl succinic anhydride, and dextrins, including mixtures of these and other nongelling, cold water dispersible, colloids.

The quantity of nongellable, cold water dispersible, colloid material is generally in a range from 30 to 70% by weight of the anhydrous components of the first dispersion with the preferred range being from 31 to 65% by weight of the anhydrous components of the first dispersion.

In preferred specific embodiments of this invention, water soluble, edible plasticizer material such as, for example, glucose, sucrose, dextrose, lactose and corn syrup and mixtures thereof are employed in conjunction with the nongelling, cold water dispersible colloid material for the purpose of increasing the stability and cold water dispersibility of the final product as well as to minimize case hardening of the finely divided particles after they have been formed. The quantity of plasticizer material should be

just enough to give adequate plasticization and beadlet stability inasmuch as during the water extraction step the risk or ease of first dispersion droplet coalescence and the extraction of fat-soluble vitamin-active composition from the droplets and particles of the first dispersion tend to increase as the plasticizer concentration is increased. A quantity of plasticizer material of 40% by weight of the anhydrous components of the first dispersion is generally the upper limit that can be employed without encountering serious difficulties with stickiness of the particles and inability to maintain fluidization of the particles in the final air drying of the particles of product obtained in accordance with the practice of the process of this invention. Indeed, beyond 40% it has been found practically impossible to desiccate the partially dehydrated, separated beadlets of first dispersion without incurring almost complete agglomeration of the beadlet mass. In general, the optimum plasticizer concentration is in the range from 15 to 40% by weight of the anhydrous components of the first dispersion with the preferred range being from 17 to 33% by weight of the anhydrous components of the first dispersion.

The quantity of water that is employed to make up the first dispersion depends in general on the particle size range desired in the final product and is dependent on the relative concentrations of nongelling, cold water dispersible, colloid material, edible plasticizer material and the fat-soluble vitamin-active composition in the final product. In general, a quantity of water in a range from 37 to 55% by weight of the first dispersion has given satisfactory results.

The water insoluble, fat-soluble vitamin-active composition consists essentially of at least one water insoluble, fat-soluble vitamin-active compound. The compound can be present in pure condition, in solution in edible oil or suspended in oil. Examples of such a compound include the vitamin A compounds such as vitamin A aldehyde, vitamin A alcohol, vitamin A esters such as vitamin A acetate and vitamin A palmitate, and the vitamin A precursor beta carotene (also called provitamin A), vitamin D compounds such as calciferol, vitamin D_2, vitamin K compounds, and vitamin E compounds such as α-tocopherol and α-tocopheryl esters such as α-tocopheryl acetate. The water insoluble fat-soluble vitamin active composition can be all liquid (in which case the first dispersion is an emulsion), all solid, but finely divided, or a mixture of finely divided solids and liquid. Where solids are involved they should be 10 microns or less in average diameter.

The water insoluble, fat-soluble vitamin-active composition, in addition to at least one water insoluble, fat-soluble vitamin-active compound generally comprises antioxidant material. Examples of such antioxidant material include butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA), tocopherol, propyl gallate and citric acid, as well as mixtures thereof. The concentration of the antioxidant material used is sufficient to impart suitable antioxidant stability to the fat-soluble vitamin-active content of the water insoluble, fat-soluble vitamin-active composition.

The quantity of water insoluble, fat-soluble vitamin-active composition depends in general on what is desired in the end product. In preferred embodiments of the invention, the vitamin A material present is generally at a concentration sufficient to provide up to 600,000 units of vitamin A activity per gram of end product, the beta carotene present is generally at a concentration sufficient to furnish up to 90,000 units of carotene potency per gram of end product and the vitamin E material present is generally at a concentration sufficient to provide up to 500 I.U. of vitamin E activity per gram of end product. In general, the concentration of the fat-soluble vitamin-active composition is in a range from 10 to 40% by weight of the anhydrous components of the first dispersion.

The water immiscible dispersing medium for making the second dispersion can be any suitable water immiscible liquid with viscosity properties like those of mineral oil. Indeed, a preferred water immiscible liquid is mineral oil. The volume of water immiscible, dispersing liquid is generally in a range from 1 to 10 volumes per volume of first dispersion, although quantities greater than 10 and quantities less than 1 can be employed.

The water extracting agent consists essentially of at least one compound which is liquid under the conditions of use under the concepts of this invention, and which extracts water from the droplets of the first dispersion without dissolving the nongelling, cold water dispersible colloid material. Examples of suitable water extracting agents include methanol, ethanol, isopropanol, acetone, methyl ethyl ketone, 1,4-dioxan and pyridine and mixtures thereof. It is preferred to employ a water extracting agent which does not extract the water insoluble, fat-soluble vitamin-active composition from the droplets of the first dispersion. So far, however, a practical water extracting agent having such a property has not been found.

The volume of water extracting agent employed under the concepts of this invention must be sufficient to effect a rapid dehydration of the first dispersion and yet be insufficient to result in substantial removal of water insoluble, fat-soluble vitamin-active composition from the droplets of the first dispersion. A preferred dehydrating agent con-

sists essentially of isopropanol and in this case a quantity of isopropanol in a range from 2.5 to 7.5 volumes per volume of water in the first dispersion has resulted in satisfactory dehydration in five minutes with a water insoluble, fat-soluble vitamin-active composition removal usually less than about 3% of the quantity of water insoluble, fat-soluble vitamin-active composition incorporated into the first dispersion.

In preferred embodiments of this invention wherein lecithin is incorporated into the second dispersion, the quantity of lecithin depends on the water concentration of the first dispersion and the weight ratio of the dispersing medium to the first dispersion. In general a quantity of lecithin in a range from 0.05 to 0.2% by weight of the first dispersion gives satisfactory results. For instance, a lecithin concentration of 0.1% of the first dispersion was effective in laboratory experiments wherein the first dispersion water concentration was in a range from 45 to 50% by weight of the first dispersion and a weight ratio of dispersing medium (mineral oil) to first dispersion was 4.

In the practice of the process of this invention the colloidal solution of cold water dispersible, non-gelling colloid material e.g. gum acacia, plasticizer when used, and water is generally made by admixing the components at 20 to 25°C. However, greater and lesser temperatures can be employed.

The second step of the process, dispersing in the colloidal solution the water insoluble, fat-soluble vitamin-active composition, is performed by admixing at 20 to 25°C. the colloidal solution and vitamin composition with vigorous agitation until the vitamin composition has been dispersed to the desired degree. A suitable degree of dispersion is obtained in the case where the water insoluble, fat-soluble vitamin-active composition has a liquid component when the droplets of the liquid component average 10 microns in diameter or less, and in the case where the vitamin composition has a finely divided solid component when the solid particles are substantially all dispersed. Although 20 to 25°C. is generally satisfactory for carrying out this step greater and lesser temperatures can be employed. Thus, there is obtained the first dispersion.

The third step of the process of this invention, that is, dispersing the first dispersion in the water immiscible dispersing medium is generally performed by admixing with vigorous agitation at a temperature in a range from 20 to 70°C. the first dispersion and the water immiscible dispersing medium. In a preferred embodiment of the process of the invention the first dispersion is heated to a temperature of 65°C. while the water immiscible dispersing medium is heated to a temperature of 46°C., and then the first dispersion is admixed with vigorous agitation with the water immiscible dispersing medium. The vigorous agitation is sufficient in duration and intensity to result in the average diameter of the droplets of the first dispersion in the dispersing medium being in a range from 0.005 to 0.02 inch. Thus, there is obtained a second dispersion.

The practice of the fourth step of this invention, that is, extracting at a temperature in a range from −10 to 0°C. with a water extracting agent water from the second dispersion until said colloid material solidifies, is preferably performed in situ. The second dispersion, while maintaining agitation, is first rapidly cooled to a temperature in the indicated range. A temperature in this range is essential in order to minimize coalescence of the droplets of the first dispersion upon addition of the water extraction agent. As soon as possible after the second dispersion has cooled to a temperature in this range, the water extraction agent is admixed with gentle agitation with the second dispersion, and gentle agitation is continued until sufficient water has been removed from the droplets of the first dispersion to "set" them or, stated another way, to solidify them into finely divided, solid particles.

The fifth step of the process, the separation at a temperature in a range from −10 to 10°C. of the solid particles from the water immiscible dispersing medium, is generally performed by filtration. In preferred embodiments of the process of the invention, the separated particles are preferably washed with additional water extraction agent at a temperature in the range from −10 to 0°C. and then with a solvent for the dispersing medium, the solvent also being at a temperature in a range from −10 to 10°C. It is essential to establish and maintain these low temperatures during the filtration and washing procedures in order to minimize sticking and agglomeration of one beadlet or group of beadlets to another beadlet or group of beadlets. A suitable solvent for the dispersing medium when it is mineral oil is hexane.

The sixth step of the process of this invention, the removal of substantially all residual moisture from the solid particles, is preferably performed by air drying as, for example, in an air fluidized bed of the solid particles. In preferred embodiments of the invention this step is performed by air drying the particles at an air temperature beginning at 70°F. and, as the residual moisture is removed, gradually raised to 140°F. until substantially all moisture has evaporated from the particles.

Frequently in the practice of these steps there tends to be a breakup of the first dispersion particularly during the initial phases of the in situ water extraction. This differs

from the dispersion of a water gelling colloid dispersion wherein gelatin of the dispersed droplets prior to water removal unalterably establishes the shape and size distribution of the particles of the product. In the present situation continued breakup of the first dispersion during water extraction until sufficient water is removed to "set" the droplets gives rise to the formation of a substantial quantity of deformed and unsymmetrical, dry, solid particles or beadlets as well as a wide spread in particle or beadlet sizes. In laboratory experiments attempts to retard first dispersion breakup during water extraction by reducing the agitation of the second dispersion resulted in rapid and complete coalescence of the dispersed first dispersion droplets as the viscosity of the second dispersion was reduced by the dehydration agent. Consequently, arresting first dispersion breakup and coalescence of the first dispersion droplets during water extraction is necessary in such instances. This can be done, and in preferred embodiments of this invention is done, by incorporating lecithin into the second dispersion. The lecithin stabilizes the dispersed first dispersion at the lower levels of dispersing medium viscosity and of agitation, and also maintains the sphericity of the dispersed droplets of the first dispersion during water extraction, whereby a dry end product is obtained in which substantially all of the particles are symmetrical spheres, the particles size is more uniform and the particle size distribution is desirably narrow.

Lecithin can be incorporated into the second dispersion by adding it to the first dispersion and by adding it to the water immiscible, dispersing medium prior to dispersing the first dispersion therein. In either case the lecithin results in better particle size control than with no lecithin at all, and reduction in particle deformation during the dehydration step, but the reduction of this deformation is not to the same extent as when the lecithin is added to the second dispersion at the water extraction step. In addition, incorporation of lecithin into the second dispersion by adding it to the first dispersion or to the water immiscible dispersing medium tends to induce the formation of a small, but substantial, quantity of very tiny particles which adhere to the larger particles to degrade product quality. Hence, incorporation of lecithin into the second dispersion is preferably done by adding it to the second dispersion along with the water extraction agent.

The quantity of lecithin incorporated into the second dispersion in preferred embodiments of the process of this invention is dependent on the water content of the first dispersion and the weight ratio of the water immiscible dispersing medium to the first dispersion. In general a quantity equivalent to a percentage in a range from 0.05 to 0.2% by weight of the first dispersion gives satisfactory results. In laboratory experiments a quantity of lecithin equivalent to 0.1% by weight of the first dispersion was effective where the first dispersion water content varied from 45 to 50% by weight of the first dispersion and the weight ratio of the water immiscible dispersing medium (which was mineral oil) to the first dispersion was about 4:1.

This invention is further illustrated by the following examples which include examples of specific embodiments of the process of this invention.

Example 1

This example illustrates the practice of a specific embodiment of the process of this invention, whereby a vitamin A product with acacia as the carrier is obtained. The potency of the product is substantially 269,700 units of Vitamin A per gram of product.

233 parts by weight of gum acacia, 58.3 parts by weight of glucose and 330.3 parts by weight of water are admixed at 20 to 25°C., whereby a colloidal solution is formed.

63.8 parts of a vitamin A palmitate concentrate having a potency of about 1,600,000 units of vitamin A activity per gram of concentrate and 2.6 parts of BHT are admixed, forming thereby a water insoluble, vitamin A composition.

The water insoluble, vitamin A composition is admixed with the colloidal solution and the mixture thus obtained is vigorously agitated until the water insoluble portion of the mixture is dispersed into droplets approximately 1 micron in diameter. There is thus obtained a first dispersion.

The first dispersion is heated to 65°C. while a quantity of mineral oil equal to about 2 volumes of the first dispersion is heated to 46°C.

The first dispersion is then admixed with vigorous mechanical stirring with the mineral oil and vigorous stirring is continued until the droplet size of the first dispersion droplets is in a range from 0.005 to 0.02 inch. Under laboratory conditions wherein the volume of the second dispersion is about 2 litres, suitable equipment for performing this step comprise a 5 litre beaker and a 4 inch diameter Cowles dissolver blade rotating at 3,000 rpm. Thus, there is obtained a second dispersion.

The second dispersion is rapidly cooled to 0°C.

About 706 parts by weight of isopropanol at −10°C. and 0.7 part by weight of lecithin dissolved in mineral oil, the quantity of which is about 20 volumes per volume of the leci-

thin are admixed with gentle agitation with the second dispersion. Under the laboratory conditions indicated in conjunction with the step of making the second dispersion, a suitable speed of the Cowles dissolver blade is about 1500 rpm. Gentle agitation of the second dispersion with the isopropanol and lecithin is continued for about 5 minutes. During this period of time water is extracted from the droplets of dispersed first dispersion, whereby they are partially dehydrated, and they solidify, forming solid particles or beadlets.

The solidified, partially dehydrated beadlets are separated by filtration from the mineral oil-isopropanol mixture, washed with about 390 parts by weight of isopropanol at −10°C. and then washed three times with chilled hexane, each hexane wash involving about 333 parts by weight of hexane at about 10°C.

The washed beadlets are then transferred to an air dryer of the fluidized bed type, wherein and whereby dehydration of the beadlets is continued with 20 to 25°C. compressed air until the moisture content of the beadlets is about 1.8% by weight of the beadlets.

The product thus obtained typically has a potency of substantially 269,700 units of vitamin A per gram of product. Vitamin loss in the practice of this embodiment typically is about 1% by weight of the original vitamin input. The product typically has a bulk density of 51.6 pounds per cubic foot and a particle size distribution such that 91.6% by weight of the particles are −30 mesh +120 mesh, U.S. screen sizes. The product rapidly disperses in water at a temperature close to freezing.

Example 2

This example illustrates the practice of a specific embodiment of this invention without the incorporation of lecithin into the second dispersion.

The steps and conditions described in Example 1 are followed, except that lecithin is not incorporated into the second dispersion.

The final product thus obtained typically has a potency of substantially 262,000 units of vitamin A per gram of product. The beadlets typically comprise many deformed particles which are dumb-bell or cruller shaped. Typically, the particle size distribution of the final product is such that 34% by weight of the product is +30 mesh, U.S. screen size and about 62.5% by weight of the product is −30 mesh +120 mesh, U.S. screen sizes.

Example 3

This example illustrates the practice of a specific embodiment of this invention, wherein lecithin is added to the water immiscible dispersing medium prior to dispersing therein the first dispersion.

The conditions and the steps of the specific embodiment of Example 1 are followed except that the 0.7 part by weight of lecithin dissolved in about 20 volumes of mineral oil per volume of lechithin is added to the mineral oil dispersing medium prior to admixing therewith the first dispersion.

The final product thus obtained typically assays substantially 257,500 units of vitamin A activity per gram of product. The product typically comprises a substantial quantity of free flowing, spherical beadlets appreciably aggregated with relatively very small beadlets. After blending the final product with 0.5% by weight of the final product of an antistatic agent such as very finely divided sodium aluminium silicate, for the purpose of separating fines from the final product, and then screening or sieving the final product, it is typical for the −120 mesh, U.S. screen size, beadlets to represent about 20% by weight of the product and the −30 mesh +120 mesh, U.S. screen sizes, fraction to be about 80% by weight of the product. A typical bulk density of the −30 mesh +120 mesh, U.S. screen sizes, fraction is 40.3 pounds per cubic foot.

Example 4

This example illustrates the practice of a specific embodiment of the process of this invention, whereby there is obtained a dry, free flowing, finely divided, vitamin E product based on acacia as the carrier.

100 parts by weight of acacia, 18.5 parts by weight of glucose and 175 parts by weight of water are admixed at 20 to 25°C., whereby a colloidal solution is obtained.

66.5 parts by weight of an α-tocopheryl acetate concentrate having a potency of substantially 1360 I.U. of vitamin E per gram of concentrate are admixed with vigorous agitation with the colloidal solution. Vigorous agitation is continued until the oil phase is dispersed into droplets approximately 1 micron in diameter. The result is a first dispersion.

The first dispersion is heated to 65°C. while approximately 2 volumes of mineral oil per volume of first dispersion are heated to 46°C. The first dispersion is then admixed with vigorous agitation with the mineral oil and the vigorous agitation is continued until the droplets of first dispersion are substantially all in a range from 0.005 to 0.02 inch. Thus, a second dispersion is formed.

The second dispersion is rapidly cooled to 0°C.

706 parts by weight of isopropanol at −10°C. and 0.7 part by weight of lecithin dissolved in a quantity of mineral oil equal to about 20 volumes per volume of lecithin are together admixed with gentle agitation

with the second dispersion, and gentle agitation is continued for 5 minutes. During this time, the dispersed droplets of the first dispersion become partially dehydrated and solidify, forming solid particles or beadlets.

The beadlets are separated from the mineral oil-isopropanol mixture by filtration, washed with about 392 parts by weight of isopropanol at −10°C. and then washed three times with hexane, at 10°C., the quantity of hexane in each wash being about 330 parts by weight.

The washed beadlets are then transferred to a dryer of the fluidized bed type, wherein dehydration is continued with 20 to 25°C. compressed air until the moisure content of the beadlets is about 1.8% by weight of the beadlets.

The resulting product is a dry, free flowing, finely divided, vitamin E product having a typical potency of 448 I.U. of vitamin E per gram of product. The particle size distribution of the product typically is such that 91.5% by weight of the product is −30 mesh +120 mesh, U.S. screen sizes.

The product readily disperses in water at temperatures close to freezing.

Example 5

This example illustrates the practice of still another specific embodiment of the process of this invention whereby a beta carotene product is obtained in which acacia is the carrier.

240 parts by weight of acacia, 61.8 by weight of glucose and 347.6 parts by weight of water are admixed at 20 to 25°C., forming a colloidal solution.

61.8 parts by weight of a suspension of very finely divided beta carotene in peanut oil, the concentration of the beta carotene being about 33% by weight of the suspension, are admixed with vigorous agitation with the colloidal solution and the vigorous agitation is continued until the droplets of the oil phase are approximately 1 micron in diameter. The result is a first dispersion.

The first dispersion is heated to 65°C. while about 2 volumes per volume of first dispersion of mineral oil are heated to 46°C.

The first dispersion is then admixed with vigorous agitation with the mineral oil and agitation is continued until the droplet size of the first dispersion is in a range from 0.005 to 0.02 inch in diameter. Thus, there is formed a second dispersion.

The second dispersion is cooled to 0°C.

706 parts by weight of isopropanol at −10°C. and 0.7 part by weight of lecithin dissolved in a quantity of mineral oil equal to about 20 volumes per volume of lecithin are together admixed with gentle agitation with the second dispersion. The resulting mixture is gently agitated or stirred for about 5 minutes. During this time the droplets of second dispersion are partially dehydrated and solidify, forming solid particles or beadlets.

The beadlets are separated from the mineral oil-isopropanol mixture by filtration followed by washing with about 390 parts by weight of −10°C. isopropanol and three times with hexane at 10°C., the quantity of hexane employed in each wash being 330 parts by weight.

The beadlets are then transferred to a dryer of the fluid bed type and dehydration is continued with 20 to 25°C. compressed air until the moisture content of the beadlets is about 1.8% by weight of the beadlets.

The result is a dry, free flowing, finely divided, beta carotene product. The product typically assays 82,500 units of carotene per gram of product. It readily dissolves in water close to freezing temperature.

Thus there is provided a process for making dry, free flowing, finely divided, fat-soluble vitamin-active products from non-gelling colloids such as acacia.

WHAT WE CLAIM IS:—

1. A process for making a dry, free flowing, finely divided, fat-soluble vitamin-active product, which comprises: (1) preparing a colloidal solution of cold water dispersible, nongelling colloid material and water; (2) dispersing in said colloidal solution a water insoluble, fat-soluble vitamin-active composition, whereby a first dispersion is formed; (3) dispersing said first dispersion in a water immiscible liquid dispersing medium, whereby a second dispersion is formed; (4) extracting at a temperature in a range from −10 to 0°C. with a water extraction agent water from said second dispersion until droplets of said first dispersion solidify, whereby solid particles are formed; (5) separating at a temperature in a range from −10 to 0°C. said solid particles from said dispersing medium and water extraction agent; and (6) removing residual moisture from said solid particles.

2. A process as claimed in claim 1 in which lecithin is incorporated into said second dispersion in an amount of 0.05 to 0.2% by weight of the first dispersion.

3. A process as claimed in claim 2 in which the amount of lecithin is 0.1% by weight of the first dispersion.

4. A process as claimed in claim 2 or claim 3 in which said lecithin is added to said dispersing medium prior to dispersing therein said first dispersion.

5. A process as claimed in claim 2 or claim 3 in which said lecithin is added to said second dispersion along with said water extraction agent.

6. A process for making a dry, free flowing, finely divided, fat-soluble vitamin-active product, which comprises: (1) admixing at

20 to 25°C. cold water dispersible, nongelling colloid material, edible plasticizer material and water, whereby a colloidal solution is formed, the quantity of said colloid material being equivalent to a concentration of said colloid material in a range from 30 to 70% by weight of the anhydrous components of said first dispersion, the quantity of said edible plasticizer material being equivalent to a concentration of said edible plasticizer material in a range from 15 to 40% by weight of said anhydrous components of said first dispersion, and the quantity of water being equivalent to a concentration in a range from 37 to 55% by weight of said first dispersion; (2) admixing with vigorous agitation said colloidal solution and a water insoluble, fat-soluble vitamin-active composition and continuing said vigorous agitation until the particle size of said composition in said colloidal solution is less than 10 microns in diameter, whereby said first dispersion is formed, the concentration of said composition being in a range from 10 to 40% by weight of anhydrous components of said first dispersion; (3) admixing with vigorous agitation at a temperature in a range from 20 to 70°C. said first dispersion with a water immiscible dispersing medium, the quantity of said dispersing medium being in a range from 1 to 10 volumes per volume of said first dispersion, and continuing said agitation until the average particle size of said first dispersion in said medium is in a range from 0.005 to 0.02 inch in diameter, whereby a second dispersion is formed; (4) cooling while continuing said vigorous agitation said second dispersion to a temperature in a range from −10 to 0°C.; (5) admixing with gentle agitation a water extraction agent and lecithin with said second dispersion, the quantity of said water extraction agent being sufficient to effect rapid dehydration of said first dispersion, but yet insufficient to extract a substantial portion of the fat-soluble vitamin-active composition from said first dispersion, the quantity of said lecithin being in a range from 0.05 to 0.2% by weight of said first dispersion, whereby water is removed from said dispersed droplets of said first dispersion and said nongelling colloidal material solidifies, forming finely divided, solid particles; (6) separating at a temperature in a range from −10 to 10°C. said solid particles from said dispersing medium and dehydrating agent; and (7) removing residual mosture from said solid particles by air drying.

7. A process as claimed in claim 6 in which the cold water dispersible, nongelling colloid material is acacia, the quantity of acacia being equivalent to a concentration of acacia in a range from 31 to 65% by weight of the anhydrous components of the first dispersion.

8. A process as claimed in claim 6 or claim 7 in which the edible plasticiser material is glucose, sucrose, dextrose, lactose or corn syrup, the quantity of edible plasticiser material being equivalent to a concentration of said edible plasticiser material in a range from 17 to 33% by weight of the anhydrous components of the first dispersion.

9. A process as claimed in any one of claims 6 to 8 in which the water immiscible dispersing medium is a mineral oil.

10. A process as claimed in any one of claims 6 to 9 in which the water extraction agent is isopropanol, the quantity of isopropanol being in a range from 2.5 to 7.5 volumes per volume of water in the first dispersion.

11. A process as claimed in claims 9 and 10 in which the solid particles are separated from the mineral oil and isopropanol by filtration followed by washing said particles with isopropanol at a temperature in a range from −10 to 0°C. and washing with hexane in a range from −10 to 10°C.

12. A process as claimed in claim 11 in which residual moisture is removed from the solid particles by air drying at temperatures in a range from 70 to 140°F.

13. A process as claimed in any one of claims 1 to 12 in which the water insoluble, fat-soluble vitamin-active composition consists essentially of a vitamin A active material and antioxidant material.

14. A process as claimed in claim 13 in which the vitamin A active material consists essentially of vitamin A palmitate.

15. A process as claimed in any one of claims 1 to 12 in which the water insoluble, fat-soluble vitamin-active composition consists essentially of beta carotene.

16. A process as claimed in any one of claims 1 to 12 in which the water insoluble, fat-soluble vitamin-active composition consists essentially of vitamin E active material.

17. A process as claimed in claim 16 in which the vitamin E material consists essentially of alpha-tocopheryl acetate.

18. A process as claimed in claim 1 and substantially as hereinbefore described with reference to and as illustrated in the Examples.

19. The product of the process of any one of claims 1 to 18.

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